Serial Number: Unknown Filing Date: Herewith

Title: BIRCH BARK EXTRACT PROCESSING UTILIZING AZEOTROPIC DISTILLATION

IN THE CLAIMS

Please cancel claims 1-71 and add new claims 72-91.

- 72. A method for obtaining betulin from birch bark, the method comprising:
 - (a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;
 - (b) separating the birch bark from the solvent to provide a first extract;
 - (c) contacting the first extract with an aqueous base to provide a second mixture;
- (d) heating the second mixture in solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective to distill off water present in the second mixture, thereby providing a third mixture;
 - (e) separating solids from the third mixture to provide a fourth mixture;
 - (f) contacting the fourth mixture with a binder to provide a fifth mixture;
 - (g) separating solids from the fifth mixture to provide a mother liquor; and
- (h) concentrating the mother liquor, precipitating betulin from the mother liquor, crystallizing betulin from the mother liquor, or a combination thereof, to provide the betulin.
- 73. The method of claim 72 wherein the betulin is obtained in a purity of at least about 95 wt.%.
- 74. The method of claim 72 wherein the mother liquor comprises lupeol.
- 75. The method of claim 72 wherein, in step (e), the solids separated from the third mixture comprise betulinic acid.
- 76. The method of claim 72 wherein the birch bark employed comprises inner birch bark.
- 77. The method of claim 72 wherein the birch bark employed comprises outer birch bark.

PRELIMINARY AMENDMENT

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78. The method of claim 72 wherein the birch bark employed comprises *Betula papyrifera*, *Betula pendula*, or a combination thereof.

- 79. The method of claim 72 wherein the aromatic hydrocarbon solvent is substituted with one to six (C_1-C_6) alkyl, halo, or trihalomethyl groups.
- 80. The method of claim 72 wherein the aromatic hydrocarbon solvent is xylenes, o-xylene, m-xylene, p-xylene, toluene, benzene, or a combination thereof.
- 81. The method of claim 72 wherein the solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C, in step (d) comprises at least one of xylene, toluene, and benzene.
- 82. The method of claim 72 wherein the contacting in step (a) further comprises heating the first mixture above about 90 °C and the separating in step (b) further comprises separating the birch bark from the solvent above about 70 °C.
- 83. The method of claim 72 further comprising, after step (b), concentrating the first extract.
- 84. The method of claim 72 wherein the fourth mixture comprises a binder selected from the group of metal hydrides, metal alcoholates, ortho-esters and dialkoxysulfates, and combinations thereof.
- 85. A method for obtaining lupeol from birch bark, the method comprising:
 - (a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;
 - (b) separating the birch bark from the solvent to provide a first extract;
 - (c) contacting the first extract with an aqueous base to provide a second mixture;
- (d) heating the second mixture in a solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective

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to distill off water present in the second mixture, thereby providing a third mixture;

- (e) separating solids from the third mixture to provide a fourth mixture;
- (f) contacting the fourth mixture with a binder to provide a fifth mixture;
- (g) separating solids from the fifth mixture to provide a mother liquor;
- (h) concentrating the mother liquor to provide crude lupeol;
- (i) washing the crude lupeol with a polar organic solvent;
- (i) recrystallizing the crude lupeol from a non-polar organic solvent; and
- (k) recrystallizing the crude lupeol from a polar organic solvent, to provide the lupeol.
- 86. The method of claim 85, wherein the polar organic solvent in (i) comprises acetone, methyl ethyl ketone, ethyl acetate, or any combination thereof.
- 87. The method of claim 85, wherein the non-polar organic solvent in (j) comprises cyclohexane, hexane, hexane, hexanes, toluene, benzene, p-xylene, m-xylene, o-xylene, trifluoromethylbenzene, or a combination thereof.
- 88. The method of claim 85, wherein the polar organic solvent in (k) comprises acetone, methyl ethyl ketone, ethyl acetate, methanol, ethanol, or a combination thereof.
- 89. A method for obtaining betulinic acid from birch bark, the method comprising:
 - (a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;
 - (b) separating the birch bark from the solvent to provide a first extract;
 - (c) contacting the first extract with an aqueous base to provide a second mixture;
- (d) heating the second mixture in a solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective to distill off water present in the second mixture, thereby providing a third mixture;
 - (e) separating solids from the third mixture;
 - (f) washing the solids with water;
 - (g) neutralizing or acidifying the solids in an aqueous acid, thereby providing a fourth

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mixture;

- (h) separating betulinic acid solids from the fourth mixture;
- (i) crystallizing the betulinic acid with a polar organic solvent; and
- (j) optionally drying the betulinic acid.
- The method of claim 89, wherein the acid in (g) comprises H₂SO₄, HCl, H₃PO₄, HNO₃, 90. HNO₂, H₃PO₃, CH₃CO₂H, CF₃CO₂H, H₃SO₃, or a combination thereof.
- The method of claim 89, wherein the polar organic solvent in (i) comprises CH₃OH, 91. EtOH, PrOH, i-PrOH, BuOH, t-BuOH, sec-BuOH, C₅H₁₁OH, acetone, ethyl acetate, methyl ethyl ketone, diethyl ketone, or a combination thereof.